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<b>(21) International Application Number:</b> PCT/GB97/02655  <b>(22) International Filing Date:</b> 26 September 1997 (26.09.97)  <b>(30) Priority Data:</b> 9620294.0                      28 September 1996 (28.09.96)      GB 9625413.1                      6 December 1996 (06.12.96)        GB 9702886.4                      12 February 1997 (12.02.97)        GB  <b>(71) Applicant (for all designated States except US):</b> AGGLOMERATION TECHNOLOGY LIMITED [GB/GB]; Unit 7, Monkswell Park, Manse Lane, Knaresborough, North Yorkshire HG5 8NQ (GB).  <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only):</b> DUFFETT, William [GB/GB]; 9 Calderdale Close, Knaresborough, North Yorkshire HG5 0DY (GB).  <b>(74) Agents:</b> SHERRARD-SMITH, Hugh et al.; Appleyard Lees, 15 Clare Road, Halifax, West Yorkshire HX1 2HY (GB).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> SPRAY CRYSTALLISED PRODUCTS AND PROCESSES  <b>(57) Abstract</b>  A method of producing a granulated product having a soft texture comprises obtaining a substance in liquid form, the substance being solid at a given temperature, atomising the liquid substance, rapidly cooling the atomised liquid so that at least partially solid particles are formed and maintaining or returning the substance at or to substantially said given temperature so that said particles agglomerate to form a soft textured product.		

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SPRAY CRYSTALLISED PRODUCTS AND PROCESSES

The present invention relates to spray crystallised products and the processes for producing such products and, in particular, to new granulated fat and other products and methods.

Cryogenic crystallisation of fats is known from EP 0393963 which describes apparatus and method for producing solid particles of fat for blending with dry ingredients such as animal feed. The method, called spray crystallisation, involves using a high pressure pump to transfer liquid fat to a jet and using compressed air to atomise the stream into a fine mist. The atomised mist passes through a laser cut ring which sprays liquid nitrogen or carbon dioxide onto the mist so that the fat particles rapidly cool to produce crystals of fat. The resultant fat crystals are mixed, for example with whey powder or flour, to give dry, lump-free powder.

Having carried out a considerable amount of research using the above technique, a range of new products and related processes have been developed. The products and processes have considerable potential and wide application particularly in the food industry but also in a diversity of other industries.

According to a first aspect of the invention there is provided a method of producing a granulated product having a soft texture comprising obtaining a substance in liquid form, the substance being solid at a given temperature, atomising the liquid substance, rapidly cooling the atomised liquid so that at least partially solid particles are formed and maintaining or returning the substance at or to substantially said given temperature so that said

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particles agglomerate to form a soft textured product.  
The method may include obtaining the substance in solid  
form and raising the temperature of the substance above  
said given temperature so that the substance melts to form  
5 a liquid.

According to a second aspect of the invention there  
is provided a soft textured product comprising an  
agglomeration of at least partially solid particles.  
10 Preferably said particles are produced by spray  
crystallisation.

According to a third aspect of the invention there is  
provided a method of converting a substance which is solid  
15 at a given temperature to a substance which is soft  
textured at said given temperature comprising obtaining  
said substance in liquid form, atomising said liquid,  
rapidly cooling the atomised liquid to form at least  
partially solid particles and agglomerating said particles  
20 to form a coherent mass.

The method may include obtaining the substance in  
solid form and melting said substance to form a liquid.  
Preferably the particles are agglomerated by maintaining  
25 or returning the particles at or to substantially said  
given temperature.

The invention further provides an agglomerated  
product having a soft texture produced using a method of  
30 the invention.

The substance may comprise a fat, oil and/or wax or  
a fat, oil and/or wax based composition. The substance  
may comprise a mixture of fats, oils and/or waxes or a  
35 mixture of fat, oil and/or wax and other components. The

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length of time for which said substance or particles are maintained at or returned to substantially said given temperature to cause agglomeration may depend on the substance used and the temperature.

5

Said given temperature may be substantially ambient or room temperature. Preferably the atomising and rapid cooling is by means of spray crystallisation. Preferably the liquid substance is transferred to a jet by means of  
10 a high pressure pump. Preferably compressed air is used to atomise the stream of liquid into a mist. Preferably the atomised mist passes through a cryogenic cooling ring, where cryogenic liquid is introduced to rapidly cool the atomised mist. Preferably cryogenic liquid is sprayed  
15 onto the atomised mist. Preferably the cryogenic liquid comprises oxygen, nitrogen, air or carbon dioxide. Preferably the rapid cooling and agglomeration results in an alteration to the crystal structure of the substance. Thus, the process of the invention alters the substance  
20 from a solid to a soft textured product.

The agglomerated product produced has several advantages due to its soft texture. For example, the product can be used in mixes without requiring further  
25 melting, it can be readily formed into shapes or packed, or can be used in, for example, bakery mixes to reduce the amount of fat used due to the more uniform smaller crystals that occur during the spray crystallisation or atomising and rapid cooling process. Thus, the product  
30 has a soft texture which allows it to be easily mixed, cut spread, moulded or otherwise handled at a given temperature whereas the unprocessed substance at a corresponding temperature is solid and consequently does not have these properties.

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According to a fourth aspect of the invention there is provided a method of producing a frozen or chilled powder product comprising obtaining a substance in liquid form, atomising the liquid substance, rapidly cooling the atomised liquid so that at least partially solid powder particles are formed and maintaining said powder at a temperature below room temperature. The method may include obtaining the substance in solid form and melting the substance to form a liquid.

Preferably said powder is stored under substantially 5°C to give a chilled powder product or under substantially -18°C to give a frozen powder product, although it will be appreciated that the storing temperature may be chosen according to the required degree of cooling and the composition of the substance.

The substance may comprise fat and/or oil such as butter, fractionated butters or cream.

The frozen or chilled powder product produced has several advantages. Due to the soft nature of the powder produced, the product can be used straight from storage at chilled or deep freeze temperatures. For example, butter or butter mixes can be produced using this method which will spread straight from either the refrigerator or the freezer. It is believed that this technical effect is due to one or both of the following effects: the modification of the crystal structure during the process; the formation of powder particles with crystallised shells and super-cooled liquid centres.

According to a fifth aspect of the invention there is provided a frozen or chilled powder product comprising a plurality of powder particles having crystallised shells

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and super-cooled liquid centres. The powder particles may comprise fat and/or oil such as butter, fractioned butters or cream.

5       The invention further provides a frozen or chilled powder product using the method of the invention.

10       The frozen or chilled powder product can be mixed with powder in various mixes, for example in baking applications, and will have the advantages of behaving as a liquid in the mix and will improve the efficiency of the fat or other substance in the baking or other process due to the change in crystal structure which occurs as the super-cooled liquid centre crystallises when the temperature rises, for example during baking.

20       The substance may comprise a substance which is solid at room temperature, typically a fat or oil based substance, and the method may include raising the temperature to melt the substance to form a liquid. Alternatively, the substance may comprise a substance which is liquid at room temperature. For example, the substance may comprise a liquid egg based substance such as yolk, albumen, whole egg and/or mixtures thereof. The substance may comprise liquid butter and/or cream or other dairy substances or products and/or mixtures thereof. Thus, these substances can be processed into a frozen or chilled powder which is stored at frozen or chilled temperatures.

30       Such base substances are normally damaged during the normal freezing process and the process of the invention minimises this damage. For example, normal freezing of a liquid substance can cause the base substance to separate

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out and the water content to freeze separately. The spray crystallisation technique of the invention inhibits this.

Furthermore, the rate at which these products can be  
5 thawed is greatly increased when converted into a powder,  
so much so that the frozen powder can be introduced into  
a blender or mixer and mixes easily as it thaws. Thawing  
of a frozen powder product of the invention typically  
takes a few minutes in contrast to several hours for a  
10 normally frozen product of this kind to thaw into a usable  
product.

Other suitable substances include liquid mixes such  
as ice cream mixes which can be processed by means of the  
15 method of the invention into a stable frozen powder. Such  
products can be consumed directly from the refrigerator or  
freezer and are sufficiently soft to consume immediately  
due to the combined effects of the crystal size and the  
supercooled liquid fractions. Alternatively the products  
20 can be thawed to liquid in very short times either at  
ambient conditions or using microwaves. Some products  
have been shown to thaw to a liquid in a matter of  
seconds.

25 The substance may comprise water so that using the  
method of the invention a frozen powder product can be  
produced which is a mixture of fine water crystals and  
supercooled liquid water. This product can be formed into  
various shapes or used for example to prepare an ice rink  
30 and the product freezes normally when the temperature  
rises in a controlled way.

Thus it can be seen that the frozen or chilled powder  
product produced can be shaped or handled more easily than

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a corresponding normally frozen product and can be caused to freeze normally by raising the temperature.

5 The substance may comprise a sugar solution which is then processed into a frozen or chilled powder. Such a product can be used to modify or improve the rate at which certain solutions crystallise.

10 Cream in the form of a frozen or chilled powder produced according to the fourth aspect of the invention or according to the fifth aspect of the invention can be further processed as follows:-

15 (i) The frozen powder may be mixed, for example using a high speed whisk, to change from frozen cream to butter and whey. The whey may be removed by passing both products over a mesh screen and the remaining solid material is butter. This process allows the raw material, 20 i.e. cream, to be stored for extended periods, which is not possible with normally chilled cream, and still be capable of processing into butter. The additional benefit of this method is the fact that butter can be produced from a 25 simple whisking operation without the use of a butter churn.

30 (ii) The frozen powder may be thawed under refrigeration i.e. at about 4 to 8°C and may then be whipped to give a comparable performance to cream that has been only chilled. Previously, frozen cream, i.e. cream frozen by known methods, would not whip without the addition of 35 stabilisers.

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According to a sixth aspect of the invention there is provided a method of producing a powder product comprising mixing a first substance which is fat, oil and/or wax based with a second substance which is not fat, oil and/or wax based to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture so that at least partially solid powder particles are formed.

The first and/or second substance may be melted or otherwise processed to form a liquid substance before, after or during the mixing. Preferably the substances are premixed at the melting stage.

Preferably the first substance comprises fat, oil and/or wax. The second substance may comprise a liquid and/or a powder and may be fat, oil and/or wax soluble. The second substance may comprise a colour, flavour and/or vitamin substance. For example, the second substance may comprise liquid flavour resins and/or emulsions, liquid colour resins and/or emulsions, liquid vitamins or vitamin emulsions, typically oil soluble vitamins. The method of the invention produces a stable powder with enhanced carrier protection against flavour and/or colour losses caused by moisture, oxygen and light. Also the release of the active constituents, for example of the second substance, can be controlled by the fat mixture used.

In the case of the second substance comprising a powder, the method provides a stable powder product in which the powder substance is encapsulated by the first substance. Levels as high as 75% powder encapsulation have been achieved. The method allows the encapsulation of a variety of powders. The second substance may comprise a chemical in powder form. The method provides encapsulation of such chemicals either singly or in

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multiples and the release of these chemicals from the stable powder product produced can be controlled by the temperature required to melt the first substance. Thus, chemicals that would normally react with each other can be prevented from so reacting by processing according to the invention and due to the absence of water.

The second substance may comprise a colour and/or flavour in powder form which can be encapsulated either singly or in multiples. The release of the colour and/or flavour from the stable powder product produced can be controlled by the temperature required to melt the first substance. Furthermore, the colours and/or flavours will have an added protection from degradation caused by moisture, oxygen and light.

The second substance may comprise an unstable powder such as vitamins which can be encapsulated either singly or in multiples using the method of the invention. The release of the unstable powders involved can then be controlled by the temperature required to melt the first substance and again the powders will have an added protection from degradation caused by moisture, oxygen and light.

The second substance may comprise one or more starches, flour and/or hydrocolloids typically in powder form with or without the addition of a suitable lecithin. The method provides encapsulation of such powders to provide a product with one or more of the following properties: the first substance coating allows these products to be mixed with hot or boiling liquids without subsequent lumping and poor mixing that normally occurs; the coating slows down the ability of the hot or boiling liquid to reach all of the thickening agent at once and

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this prevents lumps or particles of semi-dissolved or dispersed product being formed. Additionally, the emulsion formed by mixing with hot liquid has enhanced viscosity caused by hydrogen bonding between the constituents which assists the stability of viscosity when the product is vigorously mixed. Thus, the method of the invention may include subsequent mixing with a hot or boiling liquid.

10       The second substance may comprise an emulsifier and/or a preservative. In this case, the product produced by the method of the invention may be used for example in the leather industry. Typically the product will be a stable powder at room temperature and will be more  
15       convenient to use, stable and will improve the oil/fat/wax and preservative penetration into, for example, leather or the like.

20       The second substance may comprise one or more spices or spice products, either in original form, milled or not milled, or blended with other ingredients such as salt. The encapsulation of the spice prevents attack from light, heat and oxygen and greatly reduces flavour loss. Furthermore, by using a high oil or first substance  
25       temperature, typically in the region of 50 to 250°C, before or after the powders are added or the first and second substances are mixed, it is possible to reduce microbiological loading of the powder or second substance, for example of the spices or spice mixes, to an acceptable  
30       food grade level.

35       The second substance may comprise one or more dried/freeze dried microbiological organisms, e.g. bacteria, yeasts, moulds or the like which can be encapsulated either singly or in multiples using the

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method of the invention. The rapid cooling would minimise further damage to these micro-organisms and the subsequently produced powders will have an added protection from degradation caused by moisture, oxygen and  
5 light. The release of these micro-organisms is controlled by the temperature required to melt the fat or by an interaction with a suitable enzyme.

Thus the invention includes a method of encapsulating  
10 a powder substance comprising the steps of mixing the powder substance with a fat, oil and/or wax based substance to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture. The substances and method may be substantially as described  
15 above.

According to a seventh aspect of the invention there is provided a method of producing a powder product adapted to be applied to a material such as leather comprising  
20 obtaining a liquid fat, oil and/or wax, atomising the liquid and rapidly cooling the atomised liquid so that a powder product is formed which is stable at room temperature. The product gives improved penetration when applied to leather or the like.

25 The method may include mixing an emulsifier and/or a preservative with the liquid before atomisation. The method may include obtaining a fat, oil and/or wax in solid or other form and melting or otherwise processing it  
30 to form a liquid.

The invention further provides a powder product formed using a method of the invention.

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According to an eighth aspect of the invention there is provided a method of treating a material such as leather or wooden and/or plastic surfaces comprising applying a powder product of the invention to a surface of the material, for example by rubbing. The powder may or may not be electrostatically charged.

According to a ninth aspect of the invention there is provided a method of encapsulating powder particles comprising mixing powder particles with a liquid substance to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture. Preferably the liquid substance comprises fat, oil and/or wax.

According to a tenth aspect of the invention there is provided a method of producing a vitamin product comprising mixing a vitamin based substance, either in liquid or powder form, with a liquid substance to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture. Preferably the liquid substance comprises fat, oil and/or wax.

According to an eleventh aspect of the invention there is provided a method of producing a coloured and/or flavoured product comprising mixing a coloured and/or flavoured substance, either in liquid or powder form, with a liquid substance to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture. Preferably the liquid substance comprises fat, oil and/or wax.

According to a twelfth aspect of the invention there is provided a method of making chocolate comprising obtaining cocoa butter or cocoa mass in liquid form, atomising the liquid, rapidly cooling the atomised liquid

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to form a powder and adding the powder during the chocolate making process as part of the crumb making process or at the conching/tempering stage of chocolate production. This method assists in controlling and/or  
5 reducing the chocolate processing time due to the modification of the crystal structure of the cocoa butter or cocoa butter element. The invention includes chocolate made according to the above method.

10 According to a thirteenth aspect of the invention there is provided a method of producing a stable free flowing cocoa or chocolate based product comprising obtaining a cocoa or chocolate based liquid, atomising the liquid and rapidly cooling the atomised liquid to form a  
15 stable free flowing powder. The cocoa or chocolate based liquid may comprise fat and/or oil and/or mixtures such as cocoa butter, cocoa mass and various chocolate products. The product produced can be used as an ingredient in a variety of products such as in cereals, ice-cream,  
20 chocolate drinks, chilled products or the like.

According to a fourteenth aspect of the invention there is provided a method of taking spray crystallised frozen powder, which may contain for example 5-95% water  
25 content, and introducing such powder directly to a freeze drier.

The advantages would be as follows:-

30 i) The freezing of the original product would be accomplished in one rapid operation thus considerably reducing the damage caused by the normal freezing process.

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5           ii)           The product would already be in a frozen powder form which is the ideal form for freeze drying. Normally products are frozen into blocks and then broken down into irregular particles for freeze drying to commence.

10           (iii)          On occasions where the spray crystallised powder is in the form of a mixture of crystalline powders and super cooled water - this may facilitate the removal of this water more rapidly than usual and with reduced product damage during the freeze drying process.

15

The invention includes products made according to any of the above described methods of the invention.

20           The invention further provides apparatus for receiving a substance which is substantially solid at a given temperature, said apparatus comprising means for rapidly cooling said substance to produce a product which is substantially or relatively soft textured at said given temperature.

25

Preferably said apparatus comprises means for atomising a liquid substance. The apparatus may comprise means for liquifying said solid substance.

30           Preferably said substance is fat, oil and/or wax based, and preferably said soft textured product is spreadable. The substance may comprise any substance referred to herein.

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The invention further provides a product which has been at least partially solidified by spray crystallisation.

5           Preferably such product is oleaginous.

10           The steps of atomising and rapidly cooling in any of the methods described are preferably performed by conventional spray crystallisation techniques. A typical apparatus and method for performing spray crystallisation is described in EP 0393963 and the contents of that document are incorporated herein by reference. In particular, the atomisation may be by spraying and by means of an atomising nozzle through which liquid under pressure is pumped. The rate of spraying and size of the spray ejection apertures on the nozzle may be controlled or selected as desired to suit the particular requirements, for example depending on the nature of the liquid or mixture to be sprayed. The rapid cooling may be by means of directing one or more jets of cryogenic liquid, such as liquid nitrogen, oxygen, air or carbon dioxide, onto or towards the atomised spray. The size and arrangement of such jets may be controlled or selected as desired to suit the particular requirements and may be as described in EP 0393963. The methods may be controlled or automated to achieve the desired results.

30           It will be appreciated that the present invention is not intended to be restricted to the above embodiments which are described by way of example only. In particular, any features described and/or any of the steps of any method or process described may be combined in any combination and any features and/or steps described with reference to or defined by any aspect of the invention may be combined with any features and/or steps of any other

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aspect of the invention. Thus, for example, the invention includes a method according to the first or fourth aspect of the invention comprising the further step of pre-mixing as described with reference to the sixth or ninth aspect  
5 of the invention.

The reader's attention is directed to all papers and documents which are filed concurrently with or previous to this specification in connection with this application and  
10 which are open to public inspection with this specification, and the contents of all such papers and documents are incorporated herein by reference.

All of the features disclosed in this specification  
15 (including any accompanying claims, abstract and drawings), and/or all of the steps of any method or process so disclosed, may be combined in any combination, except combinations where at least some of such features and/or steps are mutually exclusive.

20 Each feature disclosed in this specification (including any accompanying claims, abstract and drawings), may be replaced by alternative features serving the same, equivalent or similar purpose, unless expressly  
25 stated otherwise. Thus, unless expressly stated otherwise, each feature disclosed is one example only of a generic series of equivalent or similar features.

The invention is not restricted to the details of the  
30 foregoing embodiment(s). The invention extends to any novel one, or any novel combination, of the features disclosed in this specification (including any accompanying claims, abstract and drawings), or to any novel one, or any novel combination, of the steps of any  
35 method or process so disclosed.

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CLAIMS

1. A method of producing a granulated product having a soft texture comprising obtaining a substance in liquid form, the substance being solid at a given temperature, atomising the liquid substance, rapidly cooling the atomised liquid so that at least partially solid particles are formed and maintaining or returning the substance at or to substantially said given temperature so that said particles agglomerate to form a soft textured product.

2. A soft textured product comprising an agglomeration of at least partially solid particles, said particles having been produced by spray crystallisation.

3. A method according to any preceding claim, wherein said particles are produced by spray crystallisation.

4. A method of converting a substance which is solid at a given temperature to a substance which is soft textured at said given temperature comprising obtaining said substance in liquid form, atomising said liquid, rapidly cooling the atomised liquid to form at least partially solid particles and agglomerating said particles to form a coherent mass.

5. A method according to any preceding claim, wherein the method includes obtaining the substance in solid form and melting said substance to form a liquid.

6. A method according to any preceding claim, wherein the particles are agglomerated by maintaining or returning the particles at or to substantially said given temperature.

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7. An agglomerated product having a soft texture produced using a method according to any preceding claim.
8. A method according to any preceding claim, wherein  
5 the substance comprises a fat, oil and/or wax or a fat, oil and/or wax based composition.
9. A method according to any preceding claim, wherein  
10 the substance comprises a mixture of fats, oils and/or waxes or a mixture of fat, oil and/or wax and other components.
10. A method according to any preceding claim, wherein  
15 the atomising and rapid cooling is by means of spray crystallisation.
11. A method according to any preceding claim, wherein  
the liquid substance is transferred to a jet by means of a high pressure pump.  
20
12. A method according to any preceding claim, wherein compressed air is used to atomise the stream of liquid into a mist.
- 25 13. A method according to claim 12, wherein the atomised mist passes through a cryogenic cooling ring, where cryogenic liquid is introduced to rapidly cool the atomised mist.
- 30 14. A method according to claim 13, wherein the cryogenic liquid is sprayed onto the atomised mist.
15. A method according to claim 13 or 14, wherein the  
35 cryogenic liquid comprises oxygen, nitrogen, air or carbon dioxide.

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16. A method according to any preceding claim, wherein the rapid cooling and agglomeration results in an alteration to the crystal structure of the substance.

5 17. A method of producing a frozen or chilled powder product comprising obtaining a substance in liquid form, atomising the liquid substance, rapidly cooling the atomised liquid so that at least partially solid powder particles are formed and maintaining said powder at a  
10 temperature below room temperature.

18. A method according to claim 17, wherein the method includes obtaining the substance in solid form and melting the substance to form a liquid.

15 19. A method according to claim 17 or 18, wherein said powder is stored under substantially 5°C to give a chilled powder product or under substantially -18°C to give a frozen powder product.

20 20. A method according to claim 17, 18 or 19, wherein the substance comprises fat and/or oil.

21. A frozen or chilled powder product comprising a  
25 plurality of powder particles having crystallised shells and super-cooled liquid centres.

22. A frozen or chilled powder product produced using a method according to any of claims 17 to 20.

30 23. A method according to any of claims 17 to 20, wherein the substance comprises a substance which is solid at room temperature and the method includes raising the temperature to melt the substance to form a liquid.

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24. A method according to any of claims 17 to 20, wherein the substance comprises a substance which is liquid at room temperature.

5 25. A method according to any of claims 17 to 20, 23 or 24, wherein the substance comprises a liquid egg based substance.

10 26. A method according to any preceding claim wherein the substance comprises liquid butter and/or cream or other dairy substances or products and/or mixtures thereof.

15 27. A method according to any of claims 17 to 20, 23 or 24, wherein the substance comprises water so that using the method of the invention a frozen powder product is produced which is a mixture of fine water crystals and supercooled liquid water.

20 28. A method according to any of claims 17 to 20, 23 or 24, wherein the substance comprises a sugar solution.

25 29. A method of producing a powder product comprising mixing a first substance which is fat, oil and/or wax based with a second substance which is not fat, oil and/or wax based to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture so that at least partially solid powder particles are formed.

30 30. A method according to claim 29, wherein the first and/or second substance is melted or otherwise processed to form a liquid substance before, after or during the mixing.

35 31. A method according to claim 29 or 30, wherein the substances are premixed at the melting stage.

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32. A method according to any of claims 29 to 31, wherein the first substance comprises fat, oil and/or wax.

33. A method according to any of claims 29 to 32, wherein  
5 the second substance comprises a liquid and/or a powder and is fat, oil and/or wax soluble.

34. A method according to any of claims 29 to 33, wherein  
10 the second substance comprises a colour, flavour and/or vitamin substance.

35. A method according to claim 34, wherein the second  
substance comprises liquid flavour resins and/or  
emulsions, liquid colour resins and/or emulsions, and/or  
15 liquid vitamins or vitamin emulsions.

36. A method according to any of claims 29 to 35, wherein  
the second substance comprises a powder and the powder  
product produced comprises the powder substance  
20 encapsulated by the first substance.

37. A method according to claim 36, wherein the second  
substance comprises a colour and/or flavour in powder form  
which is encapsulated either singly or in multiples.  
25

38. A method according to claim 36, wherein the second  
substance comprises an unstable powder such as vitamins  
which is encapsulated either singly or in multiples.

39. A method according to any of claims 29 to 36, wherein  
30 the second substance comprises one or more starches, flour and/or hydrocolloids.

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40. A method according to any of claims 29 to 36, wherein the second substance comprises an emulsifier and/or a preservative.
- 5 41. A method according to any of claims 29 to 36, wherein the second substance comprises one or more spices or spice products.
- 10 42. A method according to any of claims 29 to 36, wherein the second substance comprises one or more dried/freeze dried microbiological organisms.
- 15 43. A method of encapsulating a powder substance comprising the steps of mixing a powder substance comprising the steps of mixing the powder substance with a fat, oil and/or wax based substance to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture.
- 20 44. A method of producing a powder product adapted to be applied to a material comprising obtaining a liquid fat, oil and/or wax, atomising the liquid and rapidly cooling the atomised liquid so that a powder product is formed which is stable at room temperature.
- 25 45. A method according to claim 44, wherein the method includes mixing an emulsifier and/or a preservative with the liquid before atomisation.
- 30 46. A method according to claim 44 or 45, wherein the method includes obtaining a fat, oil and/or wax in solid or other form and melting or otherwise processing it to form a liquid.

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47. A method of treating a material comprising applying a powder product produced by a method according to any of claims 44 to 46 to a surface of the material.

5 48. A method of encapsulating powder particles comprising mixing powder particles with a liquid substance to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture.

10 49. A method according to claim 48, wherein the liquid substance comprises fat, oil and/or wax.

50. A method of producing a vitamin product comprising mixing a vitamin based substance, either in liquid or  
15 powder form, with a liquid substance to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture.

51. A method according to claim 50, wherein the liquid  
20 substance comprises fat, oil and/or wax.

52. A method of producing a coloured and/or flavoured product comprising mixing a coloured and/or flavoured substance, either in liquid or powder form, with a liquid  
25 substance to form a liquid mixture, atomising the liquid mixture and rapidly cooling the atomised mixture.

53. A method according to claim 52, wherein the liquid  
substance comprises fat, oil and/or wax.

30 54. A method of making chocolate comprising obtaining cocoa butter or cocoa mass in liquid form, atomising the liquid, rapidly cooling the atomised liquid to form a powder and adding the powder during the chocolate making

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process as part of the crumb making process or at the conching/tempering stage of chocolate production.

55. A method of producing a stable free flowing cocoa or chocolate based product comprising obtaining a cocoa or chocolate based liquid, atomising the liquid and rapidly cooling the atomised liquid to form a stable free flowing powder.
56. A method according to claim 55, wherein the cocoa or chocolate based liquid comprises fat and/or oil and/or mixtures such as cocoa butter, cocoa mass and various chocolate products.
57. A method of taking spray crystallised frozen powder and introducing such powder directly to a freeze drier.
58. Apparatus for receiving a substance which is substantially solid at a given temperature, said apparatus comprising means for rapidly cooling said substance to produce a product which is substantially or relatively soft textured at said given temperature.
59. An apparatus according to claim 58, wherein said apparatus comprises means for atomising a liquid substance.
60. An apparatus according to any claim 58 or 59, wherein the apparatus comprises means for liquifying said solid substance.
61. A product which has been at least partially solidified by spray crystallisation.

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62. A product produced using a method according to any preceding claim.

# INTERNATIONAL SEARCH REPORT

Int'l Application No  
PCT/GB 97/02655

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6    B01J2/00    B01D9/00    C11B15/00    A23C13/08    A23P1/02  
          A23L1/32    A23G1/00    A23G1/04    A23C15/14

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6    B01J    B01D    C11B    A23C    A23P    A23L    A23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 417 153 A (CHWAN-KONG KING) 23 May 1995	17, 18, 20, 22-24, 26, 27, 29-37, 40, 43-49, 58-62
A	see column 1, line 7 - column 2, line 23 see column 3, paragraph 2 see column 14, line 64 - column 17, line 23	1, 3, 5, 8-15
X	US 3 633 283 A (ABRAHAM RUDOLPH MISHKIN) 11 January 1972 see claim 1	57

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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"A" document defining the general state of the art which is not considered to be of particular relevance

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&" document member of the same patent family

Date of the actual completion of the international search

8 January 1998

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# INTERNATIONAL SEARCH REPORT

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PCT/GB 97/02655

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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